

# Document made available under the Patent Cooperation Treaty (PCT)

International application number: PCT/US05/012336

International filing date: 12 April 2005 (12.04.2005)

Document type: Certified copy of priority document

Document details: Country/Office: US  
Number: 60/563,646  
Filing date: 20 April 2004 (20.04.2004)

Date of receipt at the International Bureau: 19 September 2005 (19.09.2005)

Remark: Priority document submitted or transmitted to the International Bureau in compliance with Rule 17.1(a) or (b)



World Intellectual Property Organization (WIPO) - Geneva, Switzerland  
Organisation Mondiale de la Propriété Intellectuelle (OMPI) - Genève, Suisse

1366756

# THE UNITED STATES OF AMERICA

TO ALL TO WHOM THESE PRESENTS SHALL COME:

UNITED STATES DEPARTMENT OF COMMERCE

United States Patent and Trademark Office

*September 12, 2005*

**THIS IS TO CERTIFY THAT ANNEXED HERETO IS A TRUE COPY FROM THE RECORDS OF THE UNITED STATES PATENT AND TRADEMARK OFFICE OF THOSE PAPERS OF THE BELOW IDENTIFIED PATENT APPLICATION THAT MET THE REQUIREMENTS TO BE GRANTED A FILING DATE.**

**APPLICATION NUMBER: 60/563,646**

**FILING DATE: April 20, 2004**

**RELATED PCT APPLICATION NUMBER: PCT/US05/12336**



Certified by

Under Secretary of Commerce  
for Intellectual Property  
and Director of the United States  
Patent and Trademark Office

Please type a plus sign (+) inside this box 

PTO/SB/16 (5-03)  
Approved for use through 04/30/2003. OMB 0651-0032  
U.S. Patent and Trademark Office; U.S. DEPARTMENT OF COMMERCE  
Under the Paperwork Reduction Act of 1995, no persons are required to respond to a collection of information unless it displays a valid OMB control number.

# PROVISIONAL APPLICATION FOR PATENT COVER SHEET


This is a request for filing a PROVISIONAL APPLICATION FOR PATENT under 37 CFR 1.53(c).

PTO  
19270 U.S. PTO  
601563646

INVENTOR(S)					
Given Name (first and middle [if any])		Family Name or Surname		Residence (City and either State or Foreign Country)	
Alan Edward Victor Karol		Litke Kadziela		380 Hitchcock Road, Unit 165, Waterbury, CT 06705 410 Farmington Avenue, L-5, New Britain, CT 06053	
<input type="checkbox"/> Additional inventors are being named on the _____ separately numbered sheets attached hereto					
TITLE OF THE INVENTION (280 characters max) UV CURABLE COATING COMPOSITIONS					
Direct all correspondence to: CORRESPONDENCE ADDRESS					
<input checked="" type="checkbox"/> Customer Number		23869		<div>Place Customer Number Bar Code Label here</div>	
OR Type Customer Number here					
<input type="checkbox"/> Firm or Individual Name					
Address					
Address					
City		State		ZIP	
Country		Telephone		Fax	
ENCLOSED APPLICATION PARTS (check all that apply)					
<input checked="" type="checkbox"/> Specification		Number of Pages		24	
<input checked="" type="checkbox"/> Drawing(s)		Number of Sheets		5	
<input type="checkbox"/> Application Data Sheet. See 37 CFR 1.76		<input type="checkbox"/> CD(s), Number			
		<input type="checkbox"/> Other (specify)			
METHOD OF PAYMENT OF FILING FEES FOR THIS PROVISIONAL APPLICATION FOR PATENT (check one)					
<input checked="" type="checkbox"/> A check or money order is enclosed to cover the filing fees				FILING FEE AMOUNT (\$)	
<input checked="" type="checkbox"/> The Director is hereby authorized to charge filing fees or credit any overpayment to Deposit Account Number		08-2461		\$160.00	
<input type="checkbox"/> Payment by credit card. Form PTO-2038 is attached.					
The invention was made by an agency of the United States Government or under a contract with an agency of the United States Government.					
<input checked="" type="checkbox"/> No.					
<input type="checkbox"/> Yes, the name of the U.S. Government agency and the Government contract number are: _____					

Respectfully submitted,

SIGNATURE



TYPED or PRINTED NAME Jamie M. Larmann

TELEPHONE

(973) 331-1700

Date 4/20/04

REGISTRATION NO.

48,623

(if appropriate)

Docket Number:

500-194P

## USE ONLY FOR FILING A PROVISIONAL APPLICATION FOR PATENT

This collection of information is required by 37 CFR 1.51. The information is used by the public to file (and by the PTO to process) a provisional application. Confidentiality is governed by 35 U.S.C. 122 and 37 CFR 1.14. This collection is estimated to take 8 hours to complete, including gathering, preparing, and submitting the complete provisional application to the PTO. Time will vary depending upon the individual case. Any comments on the amount of time you require to complete this form and/or suggestions for reducing this burden, should be sent to the Chief Information Officer, U.S. Patent and Trademark Office, U.S. Department of Commerce, P.O. Box 1450, Alexandria, VA 22313-1450. DO NOT SEND FEES OR COMPLETED FORMS TO THIS ADDRESS. SEND TO: Mail Stop Provisional Application, Commissioner for Patents, P.O. Box 1450, Alexandria, VA 22313-1450.

If you need assistance in completing the form, call 1-800-PTO-9199 and select option 2.

P19LARGE/REV05

**CERTIFICATE OF MAILING BY "EXPRESS MAIL" (37 CFR 1.10)**

Applicant(s): Litke et al.

Docket No.

500-194P

Serial No.  
UnassignedFiling Date  
HerewithExaminer  
UnassignedGroup Art Unit  
Unassigned

Invention: UV CURABLE COATING COMPOSITIONS

I hereby certify that the following correspondence:

Provisional Application for Patent Cover Sheet, Patent Application (24 pgs), drawings (5 pgs)

*(Identify type of correspondence)*

is being deposited with the United States Postal Service "Express Mail Post Office to Addressee" service under 37 CFR 1.10 in an envelope addressed to: Commissioner for Patents, P.O. Box 1450, Alexandria, VA 22313-1450 on

April 20, 2004*(Date)*Barbara Thomas*(Typed or Printed Name of Person Mailing Correspondence)*  
*(Signature of Person Mailing Correspondence)*EV261768494US*("Express Mail" Mailing Label Number)***Note: Each paper must have its own certificate of mailing.**

## **UV CURABLE COATING COMPOSITIONS**

### **FIELD OF THE INVENTION**

[0001] The present invention relates to ultraviolet ("UV") curable, abrasion resistant coating compositions and methods for applying such compositions to surfaces to provide abrasion resistance. In particular, the compositions of the present invention provide coatings to surfaces, such as road reflectors, to prevent abrasion and weathering thereof. The compositions of the present invention also may be applied to filter media to provide structural rigidity thereto.

### **BACKGROUND OF THE INVENTION**

[0002] The surfaces of many molded or cast articles would benefit from showing resistance to abrasion. For instance, abrasion caused by outdoor weather conditions is a notorious cause for damage to such molded or cast articles. Conventional materials used to make molded or cast parts, however, do not typically possess sufficient surface hardness. As such, it is desirable to coat these surfaces with compositions that impart abrasion resistance thereto. For instance, the surfaces of molded road reflectors need to withstand a certain degree of abrasion due to contact from objects and weather conditions.

[0003] In addition, typical filters, such as oil filters, employ a wire screen to support the filter paper used therein because filter paper lacks rigidity. It is desirable, therefore, to coat conventional filter paper with a composition that imparts structural rigidity thereto, thereby alleviating the need for a wire screen. It also is important, however, that the coating composition not clog the filter paper.

[0004] The state of technology is not believed to provide a UV curable composition, reaction products of which having abrasion resistance to satisfy the needs of these and other applications.

### **SUMMARY OF THE INVENTION**

[0005] The present invention relates to UV curable coating compositions, which provide abrasion resistance and/or structural rigidity to surfaces. In one such aspect of the present

invention, there is provided a UV curable, abrasion resistant coating composition, which includes a curable (meth)acrylate, a photoinitiator which absorbs only in the UV range of the electromagnetic spectrum, and an inorganic filler, at least a portion of which having a particle size in the range of 1 to 1,000 nm. The particle size desirably is less than about 50 nm (diameter).

[0006] In another aspect of the present invention, there is provided a UV curable, abrasion resistant coating composition, which includes a photoinitiator which absorbs only in the UV range of the electromagnetic spectrum, the photoinitiator including 1-hydroxy-cyclohexyl-phenyl-ketone present in an amount between about 4.5% and about 5.5% by weight of the composition, trimethylolpropane triacrylate with colloidal silica nanoparticles present in an amount between about 69% and about 73% by weight of the composition, and dimethyl acrylamide present in an amount between about 22% and about 25% by weight of the composition.

[0007] In another aspect of the present invention, there is provided a UV curable, abrasion resistant coating composition, which includes a nanospheric colloidal dispersion of silica in a (meth)acrylate matrix, at least one reactive diluent, and at least one photoinitiator which absorbs only in the UV range of the electromagnetic spectrum.

[0008] In still another aspect of the present invention, there is provided an abrasion resistant coated road reflector, including a road reflector having a least one surface, the surface having a coating thereon which includes the reaction product of at least one curable (meth)acrylate, at least one photoinitiator which absorbs only in the UV range of the electromagnetic spectrum, and an inorganic filler, at least a portion of which having a particle size in the range of 1 to 1,000 nm.

[0009] In another aspect of the present invention, there is provided a road reflector having an abrasion resistant coating, the abrasion resistant coated road reflector formed by the process of providing a road reflector having at least one surface, applying a UV curable composition onto the surface, wherein the UV curable composition includes at least one

photoinitiator which absorbs only in the UV range of the electromagnetic spectrum, silica having a particle size in the range of 1 to 1,000 nm, and at least one curable (meth)acrylate, and exposing the coated surface to UV light to cure the coating composition.

**[0010]** In yet another aspect of the present invention, there is provided a filter media having structural rigidity, formed by the process of providing a filter media having a surface, applying a UV curable composition onto the surface, wherein the UV curable composition includes at least one photoinitiator which absorbs only in the UV range of the electromagnetic spectrum, silica having a particle size in the range of 1 to 1,000 nm, and at least one curable (meth)acrylate, and exposing the coated surface to UV light to cure the coating composition.

**[0011]** The present invention also is directed to methods of applying UV curable compositions to surfaces. In one such aspect, there is provided a method for applying a UV curable, abrasion resistant coating to a surface, which includes spraying a UV curable composition onto the surface to a coating thickness of about 1 to 2 mils, wherein the UV curable composition includes at least one photoinitiator which absorbs only in the UV range of the electromagnetic spectrum, silica having a particle size in the range of 1 to 1,000 nm, and at least one curable (meth)acrylate, and exposing the coated surface to UV light to cure the coating composition.

**[0012]** In accordance with another aspect of the present invention, there is provided a method of imparting abrasion resistance to a surface, including the steps of providing an article having at least one surface; applying a UV curable composition onto the surface, wherein the UV curable composition includes at least one photoinitiator which absorbs only in the UV range of the electromagnetic spectrum, silica having a particle size in the range of 1 to 1,000 nm, and at least one curable (meth)acrylate; and exposing the surface to UV light to cure the composition to an abrasion resistant coating.

**[0013]** Application of the UV curable compositions of the present invention can impart such abrasion resistance and weatherability to these surfaces. In addition, the UV curable compositions of the present invention can be applied to filter media to impart structural rigidity

thereto. The UV curable compositions of the present invention accordingly provide rigidity to filter media without compensating the permeability thereof.

### **BRIEF DESCRIPTION OF THE DRAWINGS**

[0014] Fig. 1 is a cross-sectional view of a road reflector, in accordance with an embodiment of the present invention, having an abrasion resistant coating thereon.

[0015] Fig. 2 is a perspective view of a coated filter paper of indeterminate size, in accordance with an embodiment of the present invention.

[0016] Fig. 2a is a cross sectional view of the filter paper of Fig. 2.

[0017] Fig. 3 is a graph depicting air permeability versus amount of a composition of the present invention as applied to filter media.

[0018] Fig. 4 is a graph depicting stiffness versus amount of a composition of the present invention as applied to filter media.

### **DETAILED DESCRIPTION OF THE INVENTION**

[0019] The present invention relates to UV curable, coating compositions, as well as methods for applying such compositions to surfaces, such as, for example, road reflectors. The compositions of the present invention cure by exposure to UV light to form a film coating, which shows enhanced resistance to abrasion and weathering of the surface. In other embodiments, the compositions of the present invention are applied to filter media to provide structural rigidity thereto without clogging the filter media.

[0020] The term “cure” or “curing,” as used herein, refers to a change in state, condition, and/or structure in a material that is usually, but not necessarily, induced by at least one variable, such as time, temperature, moisture, radiation, presence and quantity in such material of a curing catalyst or accelerator, or the like. The terms cover partial as well as complete curing.



[0021] The compositions of the present invention include at least one curable (meth)acrylate, at least one photoinitiator, and an inorganic filler. The photoinitiator absorbs only in the UV range of the electromagnetic spectrum. The compositions may further include a reactive diluent.

[0022] The curable (meth)acrylate component contained in the compositions of the present invention is a (meth)acrylate monomer. Examples of suitable (meth)acrylate monomers include, but are not limited to: urethane (meth)acrylates; polybutadiene (meth)acrylates; trimethylolpropane triacrylate; hexanediol diacrylate; alkoxyated hexanediol diacrylate; hydroxyl-bearing polyacrylate; hydroxyl double bond bearing polyester; tris (2-hydroxy ethyl) isocyanurate triacrylate; ethoxyated pentaerythritol tetraacrylate; and combinations thereof.

[0023] Curable (meth)acrylates, or a (meth)acrylate matrix, are present in the abrasion resistant coating compositions, for example, in an amount from about 5% to about 85% by weight in the final composition (w/w). Desirably, curable (meth)acrylates are present in an amount from about 65% to about 75% (w/w), and more desirably about 69% to about 73% (w/w).

[0024] The compositions of the present invention also include an inorganic filler. At least a portion of the filler component includes filler particles in the 1-1,000 nanometer ("nm") range. Desirably, the inorganic filler is colloidal silica nanoparticles. Such silica nanoparticles may be in a dispersion in the curable (meth)acrylate component. The filler may be present, for example, in amounts of about 30% to about 50% by weight of the (meth)acrylate component.

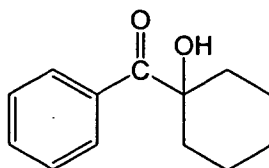
[0025] A commercially available example of such filler particles is sold under the tradename NANOCRYL, such as NANOCRYL XP 21/1045, by Hans Chemie, Germany. NANOCRYL fillers are nanosilica reinforced acrylates. In particular, NANOCRYL is a silica reinforced trifunctional acrylate monomer having a silica content of about 50% by weight. The silica component is surface-modified, synthetic SiO<sub>2</sub> nanospheres with a particle size of less than about 50 nm and a narrow particle size distribution. NANOCRYL is a colloidal dispersion of the silica nanoparticles in the curable acrylate.

[0026] Another commercially available example of filler particles is sold under the tradename HIGHLINK OG, by Clariant, France. HIGHLINK OG fillers are liquid suspensions of colloidal silica nanoparticles in an organic medium. The silica particles are spherical, non-porous, amorphous, non-agglomerated and monodispersed. The particle size ranges from about 10 nm to about 50 nm, and the silica content is about 30 to 50% by weight.

[0027] Yet another commercially available example of filler particles is sold under the tradename NANOPOX, such as NANOPOX XP 22, by Hans Chemie, Germany. NANOPOX fillers are monodisperse silica filler dispersions in epoxy resins, at a level of up to about 50% by weight. NANOPOX fillers ordinarily are believed to have a particle size of about 5 nm to about 80 nm. And NANOPOX XP 22 is reported to contain 40 weight percent of silica particles having a particle size of about 15 to 20 nm in the diglycidyl ether of bisphenol-F epoxy resin.

[0028] The abrasion resistant coating compositions of the present invention also include a photoinitiator, which absorbs only in the UV range of the electromagnetic spectrum. As such, the photoinitiator absorbs radiation at wavelengths lying outside, or shorter than, the visible light spectrum, generally less than about 400 nm. The photoinitiator responds to such UV radiation to initiate and induce curing of the curable (meth)acrylate component.

[0029] A variety of UV photoinitiators are known in the art and may be employed in accordance with the present invention. The photoinitiator may be a benzophenone or substituted benzophenone, such as, for example, an  $\alpha$ -hydroxyketone. One particularly suitable  $\alpha$ -hydroxyketone is 1-hydroxy-cyclohexyl-phenyl-ketone (commercially available as IRGACURE 184 from Ciba Specialty Chemicals, Inc.), which is represented by Ciba by the following structure:



[0030] The absorption peaks for 1-hydroxy-cyclohexyl-phenyl-ketone appear at 246, 280, and 333 nm (in methanol). In accordance therewith, an embodiment of the present invention includes a photoinitiator that absorbs radiation at wavelengths of about 333 nm or shorter.

[0031] Other suitable  $\alpha$ -hydroxyketones and blends thereof include: 2-hydroxy-2-methyl-1-phenyl-propan-1-one (absorption peaks at 245, 280, and 331 nm) (DAROCUR 1173); 2-hydroxy-1-[4-(2-hydroxyethoxy)phenyl]-2-methyl-1-propanone (absorption peaks at 276 and 331 nm) (IRGACURE 2959); and blends such as IRGACURE 1000 and IRGACURE 500 (all commercially available from Ciba Specialty Chemicals, Inc.).

[0032] Other suitable UV photoinitiators include, but are not limited to: acetophenone and substituted acetophenones; benzoin and its alkyl esters; xanthone and substituted xanthenes; diethoxy-acetophenone; benzoin methyl ether; benzoin ethyl ether; benzoin isopropyl ether; diethoxyxanthone; chloro-thio-xanthone; N-methyl diethanol-amine-benzophenone; 1-benzoyl cyclohexanol; 2-benzyl-2-(dimethylamino)-1-[4-(4-morpholinyl)phenyl]-1-butanone; amino ketones, such as IRGACURE 907, IRGACURE 369 and IRGACURE 1300 (all commercially available from Ciba Specialty Chemicals, Inc.); benzildimethyl-ketals, such as IRGACURE 651 (commercially available from Ciba Specialty Chemicals, Inc.); BAPO (bis acyl phosphine oxide) and blends thereof, such as IRGACURE 819, IRGACURE 1700, IRGACURE 1800, IRGACURE 1850, and DAROCUR 4265 (all commercially available from Ciba Specialty Chemicals, Inc.); metallocenes, such as IRGACURE 784 and IRGACURE 261 (both commercially available from Ciba Specialty Chemicals, Inc.); benzophenones, such as DAROCUR BP (commercially available from Ciba Specialty Chemicals, Inc.); and mixtures thereof.

[0033] Photoinitiators are present in the abrasion resistant coating compositions, for example, in an amount from about 1% to about 12% by weight in the final composition (w/w). Desirably, photoinitiators are present in an amount from about 3% to about 8% (w/w), and more desirably about 2.5% to about 3.5% (w/w).

[0034] The compositions of the present invention also may include a reactive diluent. A variety of reactive diluents may be employed, such as, for example, N,N-dimethyl acrylamide. Reactive diluents may be present in an amount from about 1% to about 30% by weight in the final composition (w/w). Desirably, reactive diluents are present in an amount from about 20% to about 27% (w/w), and more desirably about 22% to about 25% (w/w).

[0035] Further, the compositions of the present invention may include a light stabilizer. Light stabilizers generally prevent degradation due to UV radiation from sunlight. Light stabilizers conventionally include UV absorbers and hindered amine light stabilizers. In the compositions of the present invention, the light stabilizer desirably is free of ethylenic unsaturation, such that it will not polymerize and crosslink into the system.

[0036] Suitable light stabilizers, which are free of ethylenic unsaturation, include, but are not limited to: hydroxybenzotriazoles, such as 2-(2H-benzotriazol-2-yl)-4,6-bis(1-methyl-1-phenylethyl)phenol (available commercially as TINUVIN 900 from Ciba); hydroxyphenyltriazines, such as mixtures of 2-[4-[(2-hydroxy-3-dodecyloxypropyl)oxy]-2-hydroxyphenyl]-4,6-bis(2,4-dimethylphenyl)-1,3,5-triazine and 2-[4-[(2-hydroxy-3-tridecyloxypropyl)oxy]-2-hydroxyphenyl]-4,6-bis(2,4-dimethylphenyl)-1,3,5-triazine (available commercially as TINUVIN 400 from Ciba); and hindered amine light stabilizers, such as mixtures of bis(1,2,2,6,6-pentamethyl-4-piperidinyl)sebacate and methyl(1,2,2,6,6-pentamethyl-4-piperidinyl)sebacate (available commercially as TINUVIN 292 from Ciba).

[0037] The compositions of the present invention generally have viscosities ranging from about 5 to about 3000 cps (at 25°C). Desirably, the viscosity ranges from about 10 to about 300 cps, and more desirably about 53 to about 57 cps.

[0038] In accordance with the present invention, a desirable combination of the above components includes: trimethylolpropane triacrylate as the curable (meth)acrylate component having amorphous silica dispersed therein; N,N-dimethyl acrylamide as a reactive diluent; and 1-benzoyl cyclohexanol as the photoinitiator.

[0039] Another desirable combination of the above components includes: about 5% to about 85% w/w trimethylolpropane triacrylate as the curable (meth)acrylate component having about 20-60%, preferably 50%, colloidal silica nanoparticles dispersed therein; about 1% to about 12% w/w 1-hydroxy-cyclohexyl-phenyl-ketone as the photoinitiator; and about 1% to about 30% w/w N,N dimethyl acrylamide as a reactive diluent. The viscosity of the composition desirably ranges between about 5 and 3000 cps.

[0040] Yet another desirable combination in accordance with the present invention includes: about 65% to about 75% w/w trimethylolpropane triacrylate having about 50% colloidal silica nanoparticles dispersed therein; about 3% to about 8% w/w 1-hydroxy-cyclohexyl-phenyl-ketone; and about 20% to about 27% w/w N,N dimethyl acrylamide. The viscosity of the composition desirably ranges between about 10 and 300 cps.

[0041] A particularly desirable composition of the present invention includes: about 69% to about 73% w/w trimethylolpropane triacrylate with about 50% colloidal silica nanoparticles dispersed therein; about 2.5% to about 3.5% w/w 1-hydroxy-cyclohexyl-phenyl-ketone; and about 22% to about 25% w/w N,N dimethyl acrylamide. The viscosity of the composition desirably ranges between about 53 and 57 cps.

[0042] In accordance with the present invention, the UV curable compositions may be applied to a surface to form a coating thereon. For example, the composition may be applied to the surface of a road reflector or other similar article. In alternative embodiments, the composition may be applied to filter media paper, such as that used in oil filters, to provide rigidity thereto. Additional surfaces, however, are considered well within the scope of the present invention.

[0043] Upon exposure to UV light, the compositions of the present invention cure to a coating that imparts improved weatherability, abrasion resistance, and/or increased rigidity to the surface. For example, a road reflector **10** having an abrasion resistant coating **100** on the surface(s) **200** is shown in Fig. 1. The road reflector **10** may be made of any conventional

material known by those skilled in the art. The coating **100** improves the ability of the road reflector **10** to withstand harsh weather conditions.

[0044] In accordance with another embodiment, as depicted in Fig. 2, a filter media, e.g., filter paper **20**, is coated with a composition of the present invention. The coating **300** is applied to the surface(s) **400** of the filter paper **20** to provide structural rigidity thereto. Such rigidity allows the filter paper to be used in an apparatus, such as, but not limited to, an oil filter, without the need for a wire screen to provide support. Further, the compositions of the present invention desirably provide such rigidity without clogging the filter media.

[0045] In accordance with some embodiments of the present invention, the coatings provide abrasion resistance to a surface. The level of abrasion resistance imparted to a surface generally is determined by post-cure gloss maintained by the coating. Maintenance of post-cure gloss may be measured by subjecting the coated surface to abrasion resistance testing, such as that of Federal Specification FF-W-1825. In accordance with this test, the coated surface is subjected to a steel wool abrasion procedure. More specifically, grade 3 steel wool is placed on the coated surface. A load of about 50 lbs is applied, and the surface is rubbed about 100 times with the steel wool. The abrasion resistant coatings of the present invention desirably maintain about 90% of their post-cure gloss when subjected to this standard test, and more desirably about 95% of their post-cure gloss. The ability of the coatings to maintain such high gloss levels demonstrates their resistance to abrasive conditions.

[0046] In addition, once applied and cured, the compositions of the present invention desirably provide abrasion resistance without yellowing to a substantial degree over time. The degree to which an object color departs from its colorless or white nature toward yellow is measured by subjecting the coating to standard testing, such as ASTM E313, which is incorporated herein by reference. For instance, the coatings of the present invention desirably do not yellow more than about 40% when subjected to standard yellowness testing for 500 hours.

[0047] The present invention also is directed to methods for applying the UV curable coating compositions to a surface. According to these methods, a UV curable composition is

sprayed onto a surface to a coating thickness of about 1 to 2 mils. The UV curable composition contains at least one photoinitiator which absorbs only in the UV range, an inorganic filler, desirably silica, and at least one curable (meth)acrylate. The composition also may include a reactive diluent, as well as other components as described above. Other conventional application means, such as, but not limited to, wipe-on or dipping, may be employed to apply the coating.

[0048] Once coated, the surface is exposed to UV light to cure the composition. For example, the coated article may be placed in a conveyor apparatus for cure. The Fusion conveyor system with "H" bulbs is an example of a suitable apparatus. In such a procedure, the article is placed on a conveyor belt having a belt speed of about 15 ft/min. Several lamps are positioned in the conveyor apparatus to provide UV light for cure. The lamps may be positioned, for example, at a height of about 4 inches from the surface of the coated article. Typical irradiance levels provided by the lamps are provided in Table 1 below.

**TABLE 1**

Measurement	UV A (320-390 nm)	UV B (280-320 nm)	UV C (250-260 nm)	UV V (395-445 nm)
Peak Intensity (W/cm <sup>2</sup> )	0.589	6.00	0.082	0.36
Energy (J/cm <sup>2</sup> )	1.208	1.172	0.148	0.688

[0049] As the article proceeds through the conveyor apparatus, the coated surface is exposed to the UV light source, and the composition is cured. The cured composition imparts abrasion resistance, improved weathering properties, and/or rigidity to the surface, which enable it, for example, to withstand harsh outdoor conditions.

### **EXAMPLES**

#### **EXAMPLE 1:**

[0050] This example describes various formulations of components used to make UV curable compositions of the present invention. Table 2 depicts the weight percent of each of the components combined to form the compositions (Compositions A-G). The viscosities of

Compositions A-G are desirably between about 5 and 3000 cps. Composition G, for example, had initial viscosities at 25°C of about: 50 cps (25 rpm); 55 cps (50 rpm); and 53 cps (250 rpm).

**TABLE 2**

Component	A	B	C	D	D-1	E	F	G
Trimethylolpropane triacrylate with 50% silica	80.00	74.20	74.10	76.20	76.20	76.10	75.60	71.20
N,N-dimethyl acrylamide	20.00	18.80	18.80	18.80	18.80	18.80	18.80	23.80
1-benzoyl cyclohexanol	5.00	5.00	5.00	5.00	5.00	5.00	5.00	5.00
Diphenyl (2,4,6-trimethylbenzoyl) phosphine oxide <sup>1</sup>	2.00	2.00	2.00					
Surfactant			0.10			0.10	0.10	
Hydroxybenzotriazole <sup>2</sup>							0.50	

<sup>1</sup> Available from BASF Corporation as Lucirin® TPO

<sup>2</sup> Available from Ciba Specialty Chemicals, Inc. as Tinuvin® 900

[0051] After combining the components, the resulting Compositions A-G were applied to a surface by spraying (20 psi air pressure) to form a coating thereon. In this Example, the surfaces were those of road reflectors (white or yellow), however these compositions may be applied to a variety of other suitable surfaces. The wet coating thicknesses as applied to the white and yellow reflector surfaces (or "sides") are listed in Table 3 for each of the compositions.







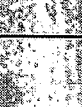




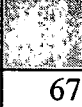

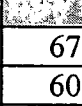


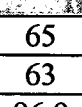
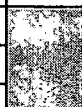



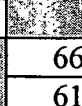











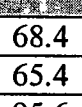







**TABLE 3**

Composition	Coating Thickness (mil)		
	White	Yellow	Side
<b>A</b>	2	1	1
	2	1.5	2
<b>B</b>	2	1.5	1
	1.5	1.5	2
<b>C</b>	1	2	1
	1.5	2	2
<b>D</b>	1.5	1	1
	2	1.5	2
<b>D-1</b>	1		1
	1.5		2
<b>E</b>	1	2	1
	1.5	1	2
<b>F</b>	1	2	1
	1.5	1	2
<b>G</b>	2		1
	1.5		2

[0052] The coated surfaces were exposed to UV light to effect curing. More specifically, the coated reflectors were placed in a Fusion conveyor apparatus having two UV lamps (fitted with "H" bulbs) positioned about four inches above the coated surfaces. The belt speeds were generally 13-15 ft/min. After exposure to the UV light, the cured coatings imparted abrasion resistance properties to the surfaces of the road reflectors.

[0053] Abrasion resistance was measured by subjecting the surfaces to standard abrasion resistance testing (Federal Specification FF-W-1825). In accordance with this procedure, gloss measurements were taken for one and/or two surfaces (sides) of the reflectors before and after abrasion with grade 3 steel wool. Table 4 lists these gloss measurements, as well as percent gloss maintained for each of Compositions A-G.

**TABLE 4**

Composition		White		Yellow	
	Gloss	Side 1	Side 2	Side 1	Side 2
<b>A</b>	initial	67		71	
	post	64		68	
	% maintained	95.5		95.8	
<b>B</b>	initial	67		73	
	post	65		66	
	% maintained	97.0		90.4	
<b>C</b>	initial	67		65	67
	post	63		65	60
	% maintained	94.0		100	89.6
<b>D</b>	initial	67	65	70	65
	post	62	63	61	64
	% maintained	92.5	96.9	87.1	98.5
<b>D-1</b>	initial	69.0	67.2		
	post	65.6	62.2		
	% maintained	95.1	92.6		
<b>E</b>	initial	67		66	
	post	63		61	
	% maintained	94.0		92.4	
<b>F</b>	initial	69		69	
	post	65		63	
	% maintained	94.2		91.3	
<b>G</b>	initial	68.6	68.4		
	post	65.3	65.4		
	% maintained	95.2	95.6		

[0054] Compositions A-G maintained about 90% or higher gloss levels after exposure to abrasion resistance testing, with several compositions maintaining nearly 95% or higher. Therefore, when applied to articles that are exposed to harsh outdoor conditions, such as road reflectors, these coatings can improve weathering properties and/or abrasion resistance.

#### **EXAMPLE 2:**

[0055] This example describes another formulation of components used to make a UV curable composition of the present invention. Table 5 depicts the weight percent of each of the components combined to form the composition (Composition H).

**TABLE 5**

Component	H
Trimethylolpropane triacrylate with 50% silica	70.00
N,N-dimethyl acrylamide	25.00
1-benzoyl cyclohexanol	5.00

[0056] The initial viscosities of Composition H at 25 °C were about: 50 cps (25 rpm); 50 cps (50 rpm); and 43 cps (250 rpm). As compared to Composition G, which contains the same components in the amounts listed in Table 2 above, Composition H has a lower viscosity due, at least in part, to the increased amount of reactive diluent (N,N-dimethyl acrylamide) present in the composition. Therefore, the viscosity of the compositions of the present invention may be altered in such manner as desired.

**EXAMPLE 3:**

[0057] This example describes another formulation of components used to make a UV curable composition in accordance with the present invention. Table 6 depicts the weight percent of each of the components combined to form the composition (Composition I).

**TABLE 6**

Component	I
Trimethylolpropane triacrylate with 50% silica	72.00
N,N-dimethyl acrylamide	25.00
1-hydroxycyclohexyl phenyl ketone	3.00

[0058] The initial viscosity of Composition I at 25°C was about 57 cps. Once applied to a surface and cured, Composition I was subjected to yellowness testing, as described above. The results were as follows:

**TABLE 7**

Time	Degree of Yellowness
Initial	3.15
500 hours	4.41
% Change	39.68

[0059] After 500 hours of standard yellowness testing, the coating of the present invention yellowed to a degree of less than about 40%.

**EXAMPLE 4:**

[0060] This example describes the application of a composition of the present invention to filter media. Composition D, as listed in Table 2 above, was used. More specifically, Composition D was sprayed onto filter paper to increase the rigidity and stiffness thereof without clogging the filter paper. In accordance with this example, one or more applications of Composition D were sprayed onto the filter paper, on one or both sides of the filter paper. The number of applications and sides to which it was applied are indicated in Table 8 below, as well as the total weight of the coating (in g) that was applied (listed as Filter Papers J-N). After application, air permeability for each coated filter paper was measured in accordance with standard permeability testing, utilizing Frazier air permeability testing equipment. The equipment measures air flow, which is expressed in terms of volumetric flow per unit area, at a set differential pressure across a sample of the media. In accordance with such testing, a significant blinding, or clogging, of pores by the composition would be represented by a corresponding reduction in air permeability. The results of the air permeability testing, which are provided as Frazier measurements (averages), also are provided in Table 8 below. These Frazier numbers represent air permeability.

**TABLE 8**

Filter Paper	Number of Applications/Sides	Coating Weight (g)	Frazier (avg)
J	1/1	0.50	63.0
K	3/1	1.24	65.9
L	2/1	1.61	62.2
M	4/1	2.77	60.0
N	2/2	3.50	59.8

[0061] A corresponding graph depicting the air permeability averages (in cfm, i.e., cubic feet per minute or the "Frazier Number") versus amount (in g) of Composition D applied is depicted in Fig. 3. This data shows that the application of Composition D to filter paper had little or no effect on air permeability. Increasing the number of applications or sides to which it was applied similarly had little effect on air permeability. The composition of the present invention provided rigidity without clogging the filter paper.

**EXAMPLE 5:**

[0062] This example also describes the application of compositions of the present invention to filter media. In accordance with this example, Composition I, the components of which are listed in Table 6 above, was sprayed onto a number of filter paper samples. The coating weight for each sample is identified in Table 9 below (Filter Papers O-S).

**TABLE 9**

<b>Filter Paper</b>	<b>Weight (g)</b>
O	1.521
P	1.271
Q	1.625
R	1.812
S	1.895

[0063] After application, stiffness for each coated filter paper was measured by subjecting the samples to standard stiffness testing using a Tabor Stiffness Tester. This equipment is used to provide accurate test measurements to +/- 1.0% in stiffness and resiliency for evaluating paper board, plastic, metal, fabric, felt, leather, wire and other sheet materials in thickness of 0.004 inches to 0.125 inches (in accordance with TAPPI T489, ASTM D5342, ASTM D5650, ASTM D747 and UNE 57-075-74). The samples were cut and stiffness was measured in both the machine direction ("MD"), which is the long axis of the filter paper, and the cross direction ("CD"). The results of the stiffness measurements (in mg) versus the amount (in g) of the coating applied are depicted in Fig. 4. As can be seen in Fig. 4, the MD stiffness values are relatively close to the stiffness of a wire screen, or about 1600 mg. As such, these coatings can provide sufficient rigidity to filter paper to replace the wire screens conventionally used in oil filters, or any other similar apparatus.

**WHAT IS CLAIMED IS:**

1. A UV curable coating composition reaction products of which are abrasion resistant, comprising:
  - at least one curable (meth)acrylate;
  - at least one photoinitiator which absorbs only in the UV range of the electromagnetic spectrum; and
  - an inorganic filler, at least a portion of which having a particle size in the range of 1 to 1,000 nm, wherein said coating is capable of maintaining about 95% of its post-cure gloss when subjected to about 100 cycles of grade 3 steel wool with a load of about 50 lbs applied per Federal Specification FF-W-1825.
2. The UV curable coating composition according to claim 1, wherein the particle size of said inorganic filler is less than about 50 nm.
3. The UV curable coating composition according to claim 1, wherein said at least one curable (meth)acrylate is selected from the group consisting of: urethane (meth)acrylates; polybutadiene (meth)acrylates; trimethylolpropane triacrylate; hexanediol diacrylate; alkoxylated hexanediol diacrylate; hydroxyl-bearing polyacrylate; hydroxyl double bond bearing polyester; tris (2-hydroxy ethyl) isocyanurate triacrylate; ethoxylated pentaerythritol tetraacrylate; and combinations thereof.
4. The UV curable coating composition according to claim 1, wherein said at least one photoinitiator comprises a benzophenone.
5. The UV curable coating composition according to claim 4, wherein said benzophenone is 1-hydroxy-cyclohexyl-phenyl-ketone.
6. The UV curable coating composition according to claim 1, wherein said at least one photoinitiator absorbs at wavelengths of about 333 nm or shorter.

7. The UV curable coating composition according to claim 1, wherein said inorganic filler comprises colloidal silica nanoparticles.
8. The UV curable coating composition according to claim 7, wherein said colloidal silica nanoparticles have a particle size of less than about 50 nm.
9. The UV curable coating composition according to claim 1, wherein said inorganic filler comprises silica particles which are spherical, non-porous, amorphous, non-agglomerated and monodispersed, said silica particles having a particle size in the range of about 10 nm to about 50 nm.
10. The UV curable coating composition according to claim 9, wherein said silica particles are present in an amount of about 30% to about 50% by weight of said composition.
11. The UV curable coating composition according to claim 1, further comprising a reactive diluent.
12. The UV curable coating composition according to claim 11, wherein said reactive diluent is N,N-dimethyl acrylamide.
13. The UV curable coating composition according to claim 1, further comprising at least one light stabilizer.
14. The UV curable coating composition according to claim 13, wherein said at least one light stabilizer is selected from the group consisting of hindered amine light stabilizers, hydroxyphenyltriazines, hydroxybenzotriazoles, and combinations thereof.
15. The UV curable coating composition according to claim 13, wherein said at least one light stabilizer is free of ethylenic unsaturation.

16. The UV curable coating composition according to claim 1, wherein said at least one photoinitiator is present in an amount of at least about 1% by weight of said composition.
17. The UV curable coating composition according to claim 1, wherein said at least one photoinitiator is present in an amount of no greater than about 12% by weight of said composition.
18. The UV curable coating composition according to claim 1, wherein said at least one curable (meth)acrylate is present in an amount of at least about 5% by weight of said composition.
19. The UV curable coating composition according to claim 1, wherein said at least one curable (meth)acrylate is present in an amount of no greater than about 85% by weight of said composition.
20. The UV curable coating composition according to claim 1, wherein said inorganic filler is present in an amount between about 30% and about 50% by weight of said curable (meth)acrylate.
21. The UV curable coating composition according to claim 11, wherein:  
said at least one curable (meth)acrylate comprises trimethylolpropane triacrylate present in an amount between about 5% and about 85% by weight of said composition; and  
said at least one reactive diluent comprises dimethyl acrylamide present in an amount between about 1% and about 30% by weight of said composition.
22. The UV curable coating composition according to claim 1, wherein the viscosity of said composition is at least about 5cps.
23. The UV curable coating composition according to claim 1, wherein the viscosity of said composition is no greater than about 3000cps.



24. A UV curable, abrasion resistant coating composition, comprising:  
a photoinitiator which absorbs only in the UV range of the electromagnetic spectrum,  
said photoinitiator comprising 1-hydroxy-cyclohexyl-phenyl-ketone present in an amount  
between about 4.5% and about 5.5% by weight of said composition;  
trimethylolpropane triacrylate with colloidal silica nanoparticles present in an amount  
between about 69% and about 73% by weight of said composition; and  
dimethyl acrylamide present in an amount between about 22% and about 25% by weight  
of said composition.
25. A UV curable, abrasion resistant coating composition, comprising:  
a nanospheric colloidal dispersion of silica in a (meth)acrylate matrix;  
at least one reactive diluent; and  
at least one photoinitiator which absorbs only in the UV range of the electromagnetic  
spectrum.
26. The UV curable coating composition according to claim 25, wherein said coating is  
capable of maintaining about 95% of its post-cure gloss when subjected to about 100 cycles of  
grade 3 steel wool with a load of about 50 lbs applied per Federal Specification FF-W-1825.
27. An abrasion resistant coated road reflector, comprising:  
a road reflector having at least one surface, said surface having a coating thereon which  
comprises the reaction product of:  
at least one curable (meth)acrylate;  
at least one photoinitiator which absorbs only in the UV range of the electromagnetic  
spectrum; and  
an inorganic filler, at least a portion of which having a particle size in the range of 1 to  
1,000 nm.

28. A road reflector having an abrasion resistant coating, the abrasion resistant coated road reflector formed by the process of:

providing a road reflector having at least one surface;  
applying a UV curable composition onto said surface,  
wherein said UV curable composition comprises at least one photoinitiator which absorbs only in the UV range of the electromagnetic spectrum, silica having a particle size in the range of 1 to 1,000 nm, and at least one curable (meth)acrylate; and  
exposing said coated surface to UV light to cure said coating composition.

29. The road reflector according to claim 28, wherein the step of applying a UV curable composition further comprises spraying said UV curable composition onto said surface to a coating thickness of about 1 to 2 mils.

30. A filter media having structural rigidity, formed by the process of:

providing a filter media having a surface;  
applying a UV curable composition onto said surface;  
wherein said UV curable composition comprises at least one photoinitiator which absorbs only in the UV range of the electromagnetic spectrum, silica having a particle size in the range of 1 to 1,000 nm, and at least one curable (meth)acrylate; and  
exposing said coated surface to UV light to cure said coating composition.

31. The filter media according to claim 30, wherein the coated filter media has a stiffness of about 1600 mg.

32. A method for applying a UV curable, abrasion resistant coating to a surface, comprising the steps of:

spraying a UV curable composition onto the surface to a coating thickness of about 1 to 2 mils,

wherein the UV curable composition comprises at least one photoinitiator which absorbs only in the UV range of the electromagnetic spectrum, silica having a particle size in the range of 1 to 1,000 nm, and at least one curable (meth)acrylate; and  
exposing the coated surface to UV light to cure the coating composition.

33. A method of imparting abrasion resistance to a surface, comprising the steps of:  
providing an article having at least one surface;  
applying a UV curable composition onto the surface,  
wherein the UV curable composition comprises at least one photoinitiator which absorbs only in the UV range of the electromagnetic spectrum, silica having a particle size in the range of 1 to 1,000 nm, and at least one curable (meth)acrylate; and  
exposing the surface to UV light to cure the composition to an abrasion resistant coating.

**UV CURABLE COATING COMPOSITIONS**

**ABSTRACT OF THE DISCLOSURE**

[0064] UV curable coating compositions and methods for applying such compositions to surfaces to provide abrasion resistance and/or structural rigidity thereto. The UV curable compositions of the present invention include at least one curable (meth)acrylate, at least one photoinitiator which absorbs only in the UV range of the electromagnetic spectrum, and an inorganic filler, at least a portion of which having a particle size in the range of 1 to 1,000 nm. The compositions may further comprise a reactive diluent.

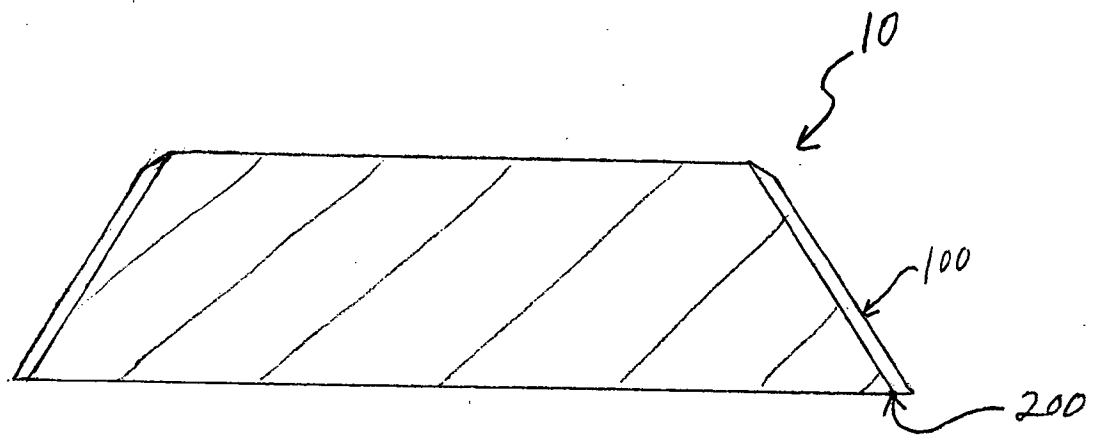


Fig. 1

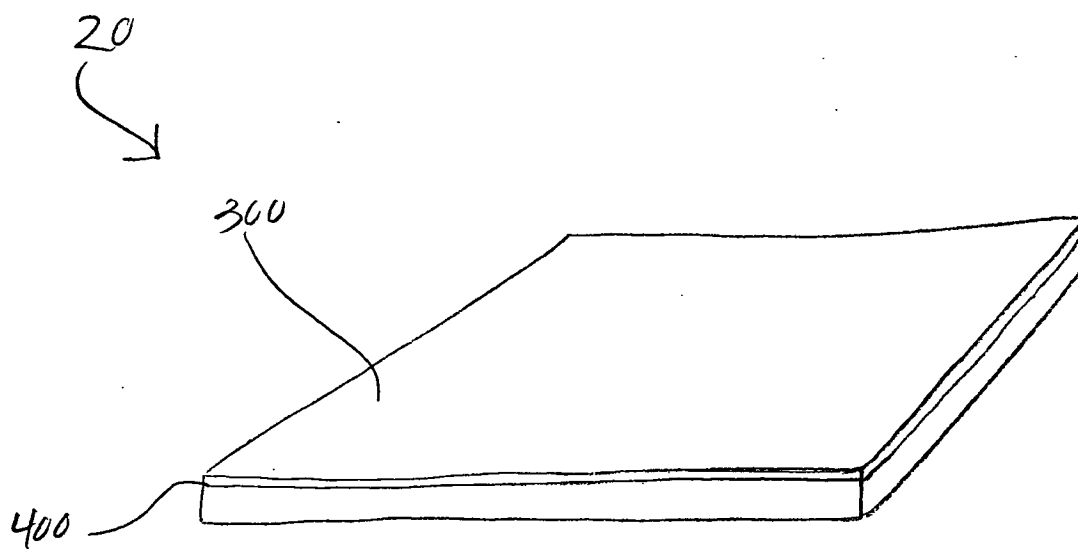


Fig. 2

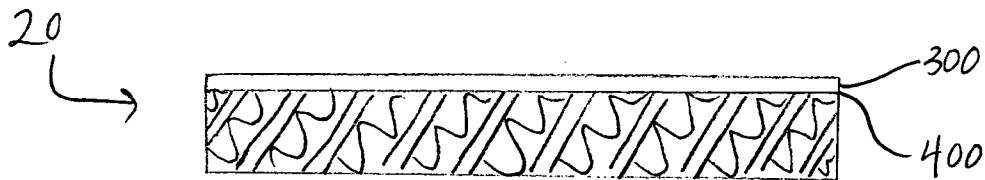


Fig. 2a

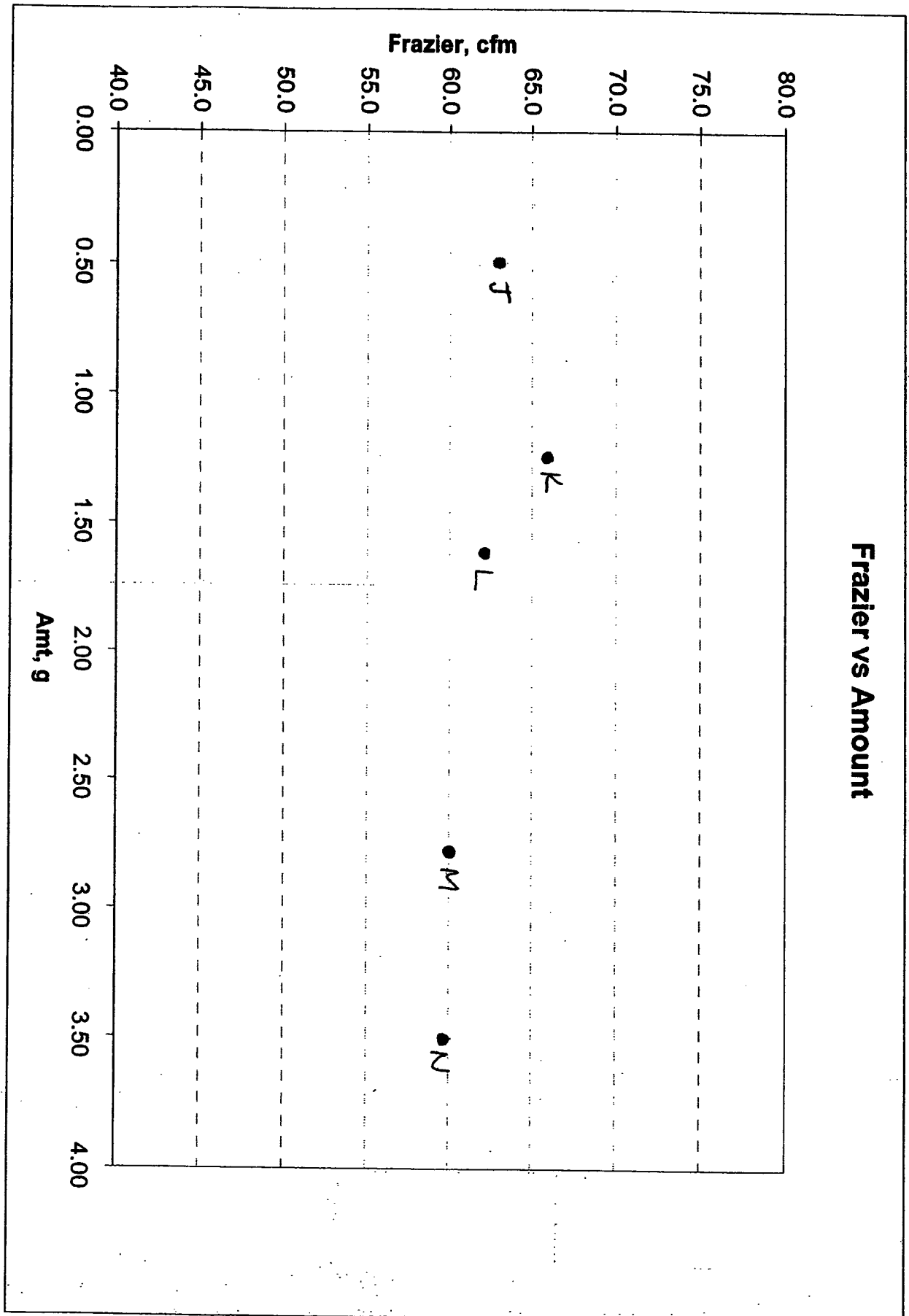


Fig. 3



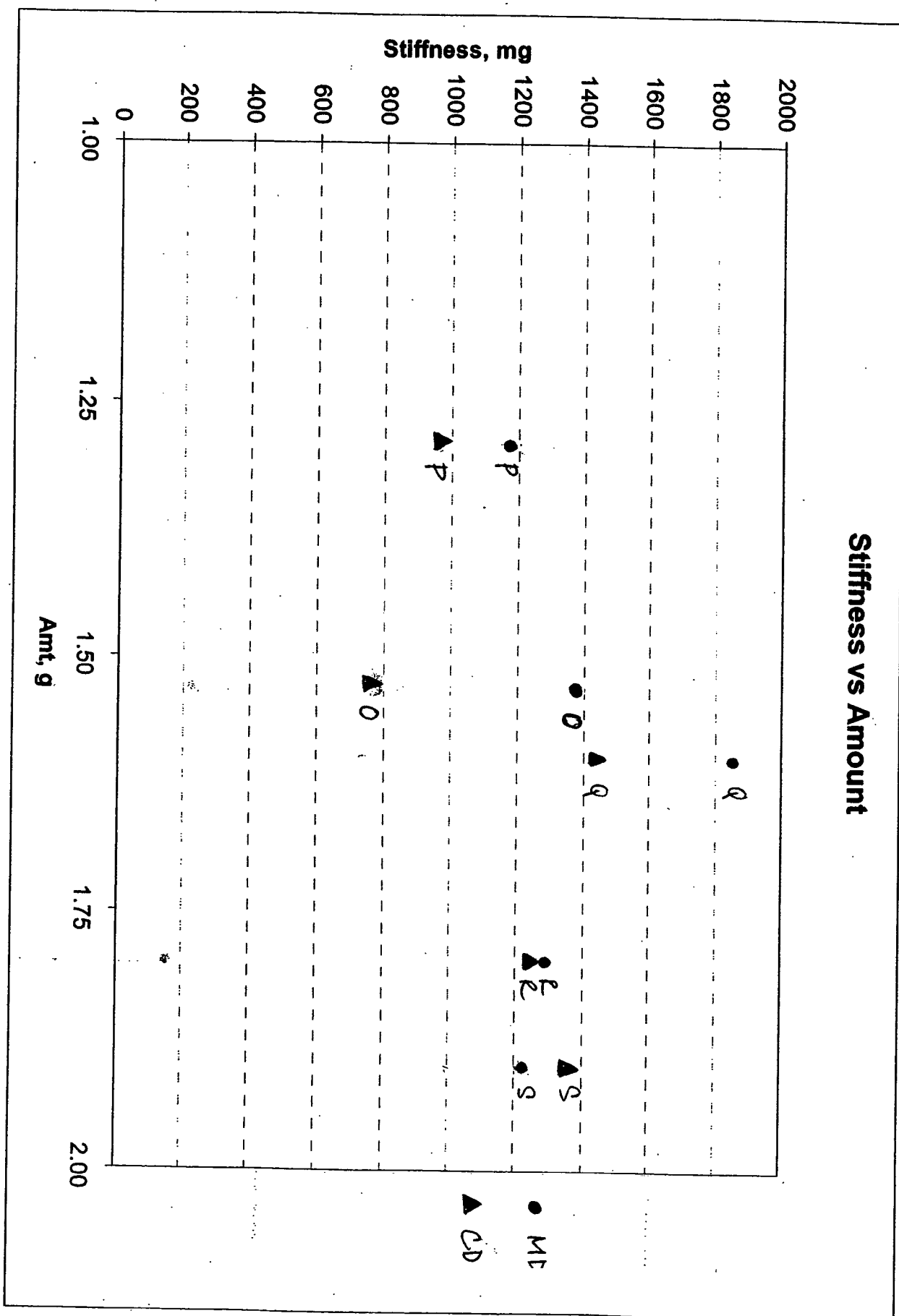


Fig. 4